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The Continuous Flow Analyzer Automation System Part I - Functional Specifications

Cincinnati Univ. Computing Center, OH

Prepared for

Environmental Monitoring and Support Lab.-Cincinnati, OH

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April 1982

THE CONTINUOUS FLOW ANALYZER AUTOMATION SYSTEM
PART I -- FUNCTIONAL SPECIFICATIONS

by

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Contract No. GS-05S-10458

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This document contains the project definition, the functional requirements, and the functional design for a proposed computer automation system for the continuous flow analyzer. The proposed system will accomplish real-time data acquisition, calibration, baseline correction, calculation of concentrations, dilution and remeasurement of offscale samples, on-line quality control checks, reagent blank subtraction, plotting of calibration curves and raw data, and bidirectional communication with the Sample File Control System. This proposed automation system will facilitate routine measurement of large numbers of samples, while providing the flexibility needed for a research environment.

Two related publications are titled, "The Continuous Flow Analyzer Automation System, Part II -- User's Guide," and "The Continuous Flow Analyzer Automation System, Part III -- Program Documentation."

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati, conducts research to:

- Develop and evaluate techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
- Investigate methods for the concentration, recovery, and identification of viruses, bacteria, and other microbiological organisms in water, and to determine the responses of aquatic organisms to water quality.
- Develop and operate an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.
- Develop and operate a computerized system for instrument automation leading to improved data collecton, analysis, and quality control.

This report was developed in the Advanced Instrumentation Section of the Environmental Monitoring and Support Laboratory in the interest of advancing laboratory techniques and quality control through computerization.

Robert L. Booth
Acting Director
Environmental Monitoring and Support
Laboratory - Cincinnati

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ABSTRACT

This document contains the project definition, the functional requirements, and the functional design for a proposed computer automation system for the continuous flow analyzer. The proposed system will accomplish real-time data acquisition, calibration, baseline correction, calculation of concentrations, dilution and remeasurement of offscale samples, on-line quality control checks, reagent blank subtraction, plotting of calibration curves and raw data, and bidirectional communication with the Sample File Control System. This proposed automation system will facilitate routine measurement of large numbers of samples, while providing the flexibility necessary for a research environment.

Two related publications are titled, "The Continuous Flow Analyzer Automation, Part II -- User's Guide" and "The Continuous Flow Analyzer Automation System, Part III -- Program Documentation."

This report was submitted in partial fulfillment of Task Assignment No. EPA-80-07 of EPA Contract No. GS-05S-10458 by the University of Cincinnati Computer Center. It covers work done between June 1, 1979 and November 16, 1979.

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Dennis Ryan of the University of Cincinnati Computer Center provided the specifications for the Sample File Control interaction.

John Teuschler and William Budde of the EPA Advanced Instrumentation Section reviewed this document and made many valuable suggestions.

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SECTION 1

INTRODUCTION

This document details the plans for a new computer automation system for the continuous flow anlayzer. The history of this project can be traced to 1974, when the Lawrence Livermore Laboratory of the University of California was first contracted by the United States Environmental Protection Agency to automate this instrument. Much pioneer work in laboratory automation was done at Livermore, and a set of automation programs written there for the continuous flow analyzer has been in use in EPA laboratories for several years.[1,2]

Recent advances in quality control procedures and laboratory data basing, however, have mandated that the automation system be updated for the 1980's. Furthermore, as the possibility of revamping the system was explored, it was decided that more general peak height determination and timing synchronization algorithms should also be developed.

Consequently, this document describes what will be a new Continuous Flow Analyzer Automation System which will communicate bidirectionally with the EPA Sample File Control System, acquire raw data from the instruments in synchronization with the automatic samplers, determine peak heights while correcting for baseline drift, calibrate, calculate concentrations, perform quality control calculations, compensate mathematically for dilution of samples, allow for subtraction of reagent blanks, print reports, and plot calibration curves and raw data.

The "Project Definition" in this document defines the scope of the project. The "Overview of the Project" provides background information needed by the system implementor. The "Requirements of the System" section is a very detailed list of what the completed system must be capable of doing. To avoid fragmenting this document, the specific algorithms and formulas to be used are included in this section, even though they are design details rather than requirements. The "Functional Design" shows the implementer's concept of how all of the required features can be incorporated into a logically organized system.

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SECTION 2

PROJECT DEFINITION

The goal of this project is to develop a computer automation system which will accept raw data from commercially available continuous flow analyzers and process the data. The system will provide for:

- bidirectional communication with the EPA Sample File Control System, so that sample backlog lists can be transferred to the automation system, and results can be transferred back to the Sample File Control System,
- real-time, unattended data acquisition from split stream continuous flow analyzers with up to three detectors,
- calibration using regression or linear interpolation,
- plotting of calibration curves during an automated run, and plotting of all raw data,
- baseline correction by subtraction of the value interpolated between blanks,
- on-line data reduction for the calculation of concentrations of samples,
- dilution and remeasurement features for offscale samples,
- on-line quality control checks, including instrument check standards, spiked samples, duplicate samples, and laboratory control standards,
- reagent blank subtraction,
- post-run manipulation of the standard calibration curve and recalculation of the concentrations of samples using stored raw data,
- detailed final reports on samples and quality control information, and
- restarting of an interrupted run.

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SECTION 3

FUNCTIONAL REQUIREMENTS - OVERVIEW OF THE PROJECT

3.1 THE NATURE OF THE INSTRUMENT

3.1.1. Scientific Principles of Continuous Flow Analyzers

Figure 1 shows the major components of a typical continuous flow analyzer. Samples* are placed in test tubes in an automatic sampler, and chemical reagents are placed in flasks. The sampler and the reagent flasks are connected by tubing to the proportioning pump, where moving rollers occlude the tubing. The resulting parastaltic action aspirates the samples and reagents into the tubing. Tubes of different inside diameters are used to control the ratios of the various fluids. In the manifold, the sample and reagent streams are merged, and the solution is mixed and sometimes heated as it passes through several glass helixes. This causes a color to develop in the solution, and the intensity of that color is directly proportional to the concentration of the analyte being measured. The stream of solution then passes through the colorimeter where a monochromatic light is directed through the fluid and onto a photodetector cell. The absorbance of the fluid is directly proportional to the saturation of the color, and a current generated by the photodetector cell is -- after being modified by electronic circuitry -- proportional to that absorbance. The electric current is transmitted to a recorder where changes in voltage are represented proportionally by movement of a pen across a graph. Consequently, the concentration of the analyte is directly proportional to the deflection of the pen on the recorder. Furthermore, a telemetry connection on the colorimeter allows a voltage proportional to concentration to be transmitted to a computer.

In some cases, detectors other than colorimeters are used to measure properties other than the color of the analytical stream. However, the resultant telemetry signal is directly proportional to concentration and can be treated similarly by a computer.

---

*In this document, the word "sample" refers to the contents of one test tube in the automatic sampler. It is a generic term which includes standards, blanks, unknowns, etc. The word "batch" denotes a complete series of samples. The word "run," when used as a noun, means the execution of the programs of the automation system.

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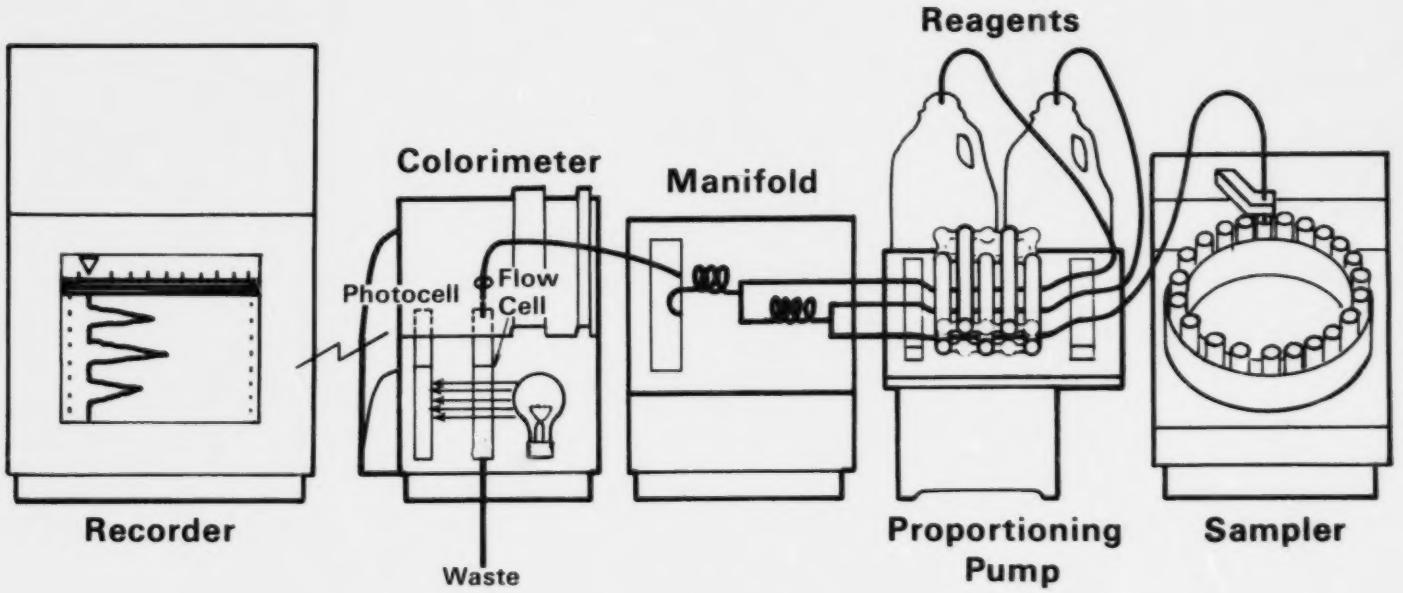

Figure 1. Typical continuous flow analyzer.

In some laboratories, a single pump aspirating samples from a single automatic sampler is used to force an analytical stream through two or three manifolds and detectors. This "split stream" processing allows for more than one determination to be made at the same time.

3.1.2 Determination of Concentration

As successive samples pass through the continuous flow analyzer, peaks are drawn on the recorder, as shown in Figure 2. The first, fifth, and ninth peaks in that figure represent distilled water blanks, and the dotted line joining the blanks is the baseline. The height of each peak is measured from the baseline to the top of the peak.

Although the peak height is proportional to the concentration of the analyte in a sample, an actual determination of the concentration of an unknown can only be made by comparing its peak height to the peak heights of a set of standards. Therefore, a batch is normally begun with a series of calibration standards which span the range of the concentrations expected in the unknowns. The peak heights of these standards are measured, and regression is then used to develop an equation relating peak height to concentration. This equation is used to calculate the concentrations of the remaining samples. Blanks are placed at regular intervals throughout a batch to monitor baseline drift. Quality control samples may be included in the batch to evaluate the accuracy and precision of the method.

3.2 INSTRUMENTS TO BE AUTOMATED

Every continuous flow analyzer to be automated by this system is a Technicon AutoAnalyzer II producing telemetry signals which are directly proportional to the concentration of the analyte. (Older Technicon Autoanalyzer I equipment which measures transmittance will not be used with this system.)

The instruments to be automated are located in the following laboratories: 1) the Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 2) the Municipal Environmental Research Laboratory, Cincinnati, Ohio, 3) the EPA Region III Surveillance and Analysis Laboratory, Annapolis, Maryland, and 4) the EPA Region V Surveillance and Analysis Laboratory, Chicago, Illinois.

3.3 NONTECHNICAL CONSIDERATIONS

3.3.1 Time and Financial Constraints

The first version of the Continuous Flow Analyzer Automation System should be ready for use by April 30, 1980. The manuscript copies of the users' guide and program documentation books should be completed by June 30, 1980. The total expense for developing these functional specifications, coding the system, and writing the documentation should not exceed $30,000.

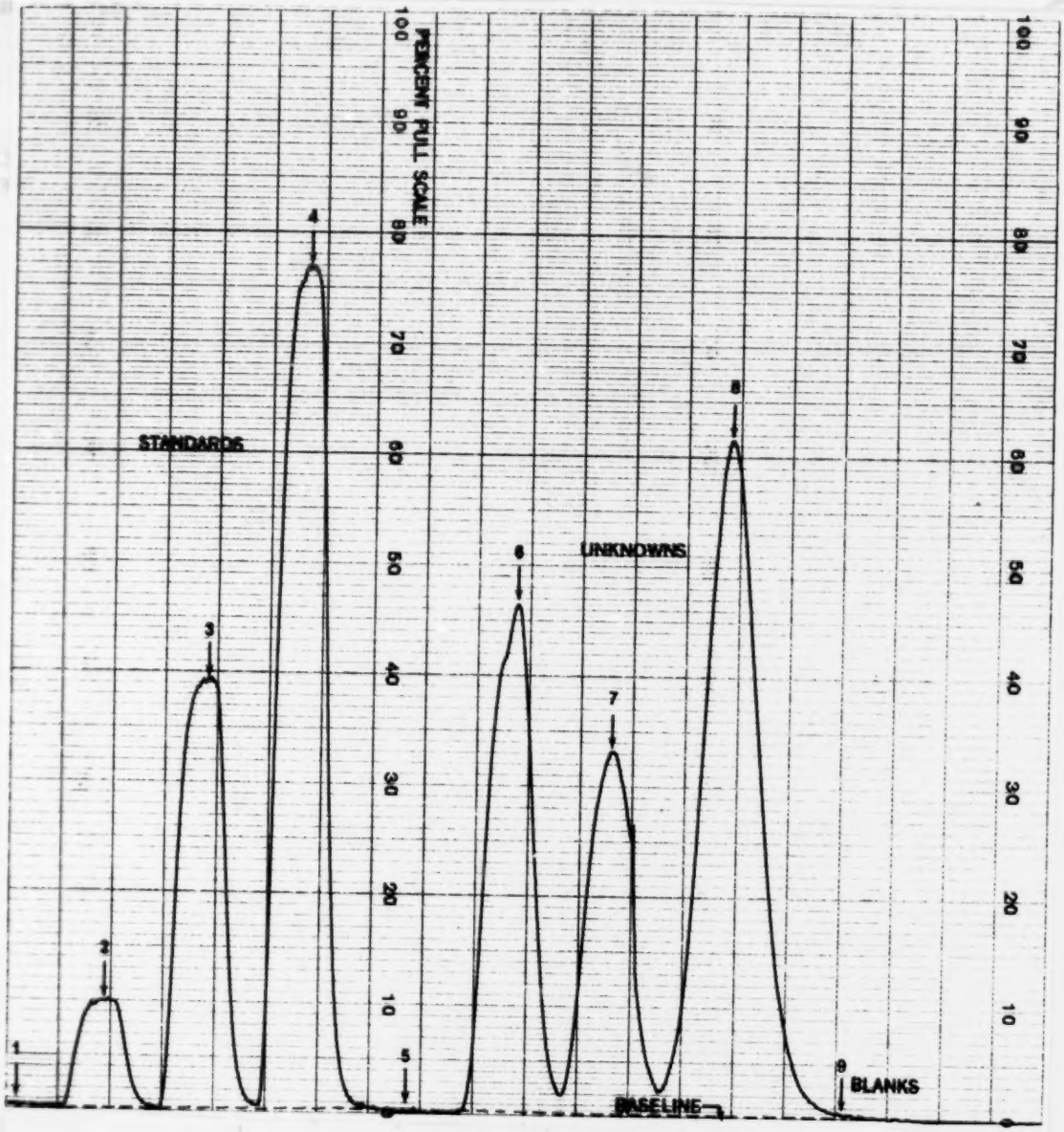

Figure 2. Strip chart recording of a typical run.

3.3.2 Existing Hardware and Software to be Utilized

The Continuous Flow Analyzer Automation System will be part of a larger laboratory automation project, and therefore many of the fundamental hardware and software decisions have already been made within the context of the larger project. Specifically, the new system will run on Data General Nova 840 computers. It will be written in Data General Extended BASIC, Revision 4.3, as enhanced by Lawrence Livermore Laboratory for real time data acquisition.[3] The operating system will be the unmodified Data General Mapped Real-Time Disk Operating System, (MRDOS) Revision 6.2, for Nova computers.

Assembly language routines for real-time data acquisition from continuous flow analyzers were written at Livermore for the original automation system, and they will be used without modification.

Custom hardware (such as interface boxes and analogue to digital converters) was designed and built for the original automation system. This hardware is currently installed and will be used without modification for the new system.

All plotting programs should be written for existing Tektronix 4000-series computer display terminals, or the hardware equivalent of such terminals.

3.3.3 Support of the Completed System

The continuous flow analyzer instruments will not be modified in any way, and they will be supported by their manufacturer.

The Data General computer, excluding the custom hardware and the terminals, will be supported by the Data General Corporation under a maintenance contract which must be maintained by the laboratory owning the computer.

All custom hardware will be supported for a minimum of one year after delivery by the Advanced Instrumentation Section, Cincinnati, Ohio. A supply of spare parts will be provided so that repairs can be made on a module swapping basis. Computer terminals will be supported primarily by their manufacturers.

The Mapped Real-Time Disk Operating System will be supported by the Data General Corporation.

The enhanced version of Extended BASIC will be supported by the Advanced Instrumentation Section for a minimum of one year after delivery.

The BASIC programs of the Continuous Flow Analyzer Automation System will be supported by the Advanced Instrumentation Section for a minimum of one year after delivery.

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All of these statements of support assume that the supported items will not be altered by unauthorized persons. Requests for enhancements to the delivered system should be brought to the attention of the Advanced Instrumentation Section. Periodically the automation system will be updated as time and personnel considerations permit. A user who wishes to make custom modifications to hardware or software must be prepared to assume support of the personalized item.

3.4 FUTURE CONSIDERATIONS

The Continuous Flow Analyzer Automation System should be designed as a modular system so that it can easily be enhanced to keep pace with advances in methodology. It must be thoroughly documented to facilitate such enhancements. If possible, it should be designed so that it can be adapted at some time in the future to receive processed data from microprocessor-based instruments.

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SECTION 4

FUNCTIONAL REQUIREMENTS - REQUIREMENTS OF THE SYSTEM

4.1 RAW DATA ACQUISITION

The telemetry signal from the continuous flow analyzer normally ranges from 0 to 10 volts, direct current. This signal will be differentially coupled at the instrument and transmitted to an analogue to digital converter which will generate an integer between 0 and 16,383 (i.e. $2^{14} - 1$). The resulting numbers will be directly proportional to the voltage of the telemetry signal, and consequently to the concentration of the analyte in the detector.

The Continuous Flow Analyzer Automation System will collect readings from the analogue to digital converter by using assembly language routines developed at Lawrence Livermore Laboratory. Enhancements made at Livermore to the Data General BASIC will allow the BASIC program of the automation system to call these assembly language routines while the multi-user nature of BASIC is maintained. The assembly language routines will provide the flexibility needed to acquire data from the analogue to digital converter. Once they have been started by calls from the BASIC programs, the assembly language routines will be activated by interrupts from a real-time clock in the computer. Upon completion of an assembly language routine, control will be returned to the BASIC program. The assembly language routines and the enhancements to BASIC have been extensively documented.[3]

The sequence of events for data acquisition will be as follows: The BASIC program will pass parameters to the assembly language routines. These user-specified parameters will include the rate at which readings are to be taken from the analogue to digital converter, the amount of time between peaks, and the threshold height for the first timing peak. The next instruction that the BASIC program gives to the assembly language routine will be to begin the search for the first peak. From this point on, the assembly language routine will collect data from the analogue to digital converter at prescribed intervals of time. The time used by the assembly language routine for each reading will be roughly 0.00005 seconds, so that no other users will be noticeably affected.

It would be impractical for the assembly language routine to actually take readings from the analogue to digital converter constantly, however peak heights can be accurately determined in continuous flow analysis from readings taken 0.5 to 1.5 seconds apart. Therefore, the assembly language routine will take only sixty readings from the analogue to digital

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converter for each peak, and these sixty readings will be evenly spaced throughout a "data acquisition window" centered on the top of the peak and having a width specified by the user.

The concept of a data acquisition window creates one additional complication, however, and that is the synchronization of the assembly language routines with the automatic sampler so that the data acquisition windows are centered on the tops of the peaks. For this reason, the first three samples of every batch will have to be timing set standards. When the BASIC program instructs the assembly language routine to begin looking for the first peak, the assembly language routine will begin to take readings from the analogue to digital converter at the rate of once every four seconds. When it first encounters a reading higher than the threshold value specified by the user, the assembly language routine will begin to take readings at the normal, user-specified rate. When the readings drop below the threshold value, the assembly language routine will stop taking readings, and it will calculate the amount of dead time which should transpire before the next data acquisition window begins. Thereafter, it will be the responsibility of the BASIC program to maintain timing synchronization by determining if the peaks are centered in the data acquisition windows and by instructing the assembly language routine to lengthen or shorten the dead time between windows, if necessary. The second and third timing set standards will give the BASIC program a chance to make fine adjustments to the timing synchronization before the important samples of the batch are dealt with.

After all of these preliminary operations are complete, the data acquisition process will become repetitive. When the BASIC program is ready for the sixty readings for the next peak, it will call the assembly language routine to provide them. Usually the BASIC program will be ready for more data long before the next sample reaches the detector. Therefore, the assembly language routine will cause the BASIC program to be suspended -- freeing more time for other users. When the assembly language program has acquired the sixty readings for the next peak, it will "wake up" the BASIC program, pass the sixty values to the BASIC program, and return control to the BASIC program.

If for any reason, the BASIC program gets behind, the assembly language routine will be capable of backlogging the data for a set number of peaks until the BASIC program can catch up. This backlogging feature will not be used except in unavoidable circumstances however, since data acquisition window adjustments cannot be made on backlogged data. At the end of the automated run, the BASIC program will instruct the assembly language routine to stop, and that routine will no longer be activated by the real-time clock.

4.2 PEAK HEIGHT DETERMINATION

Two terms must be defined before the peak height determination algorithms can be discussed. The first of these is the word "level." In Figure 3A, the peak with a broad plateau has an obvious level region, and the peak in Figure 3B can be considered instantaneously level at its

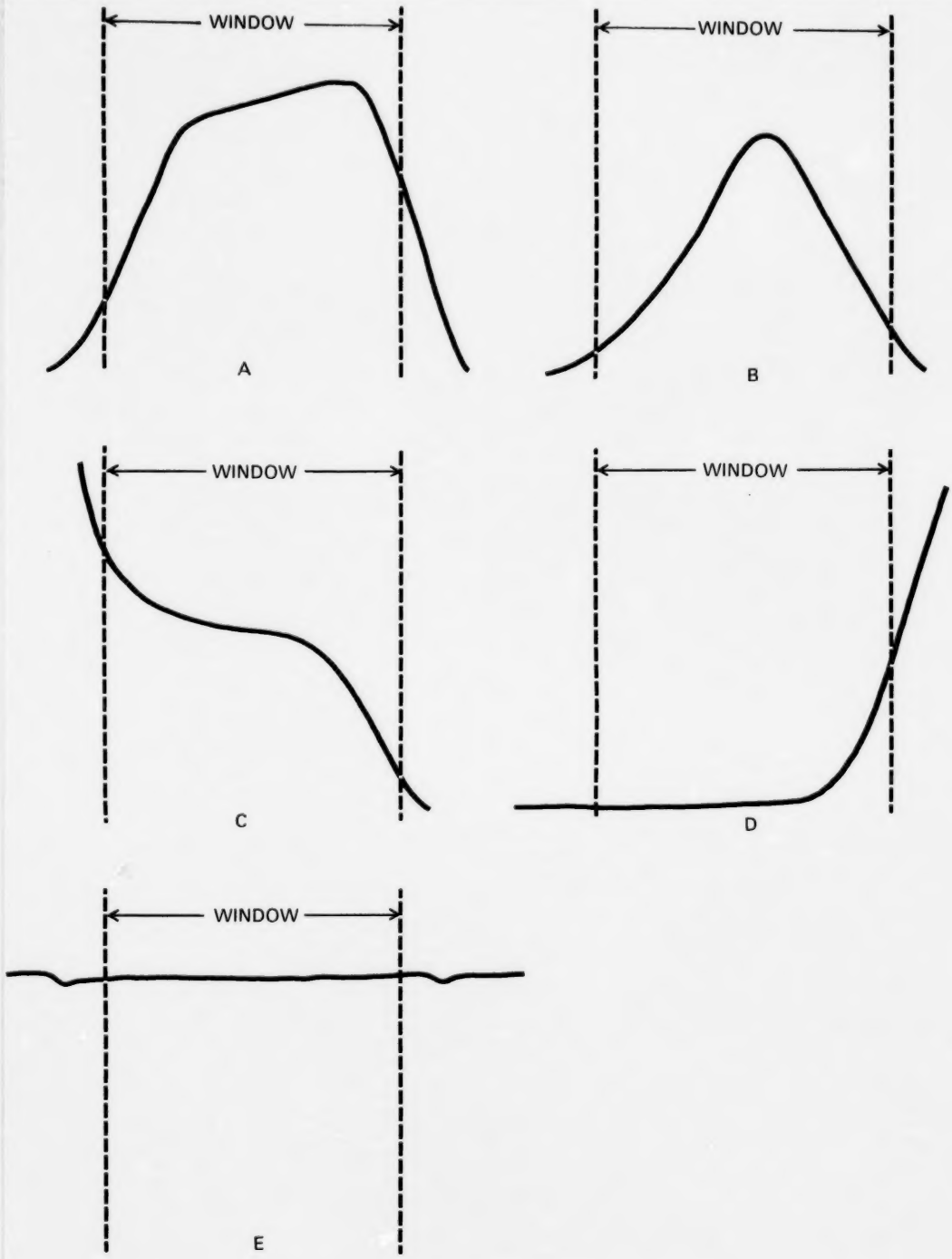

Figure 3. Types of peaks.

summit. However, a close examination of the analogue to digital converter readings for continuous flow analyzer peaks reveals that -- even for peaks with broad plateaus -- it is very unusual for any two consecutive readings to be precisely the same. Therefore, for this purpose, "level" cannot be defined simply as "having no part higher than another." Rather, reading number n of the sixty readings in the data acquisition window will be considered to be in a level place if the standard deviation of readings n-2, n-1, n, n+1, and n+2 is less than a user-specified amount.

The second term which must be defined is "smoothed value of a reading." No peak height determination should be based on a single reading from the analogue to digital converter, since that one reading may be an outlier. The Continuous Flow Analyzer Automation System will work with "smoothed values" which are determined by the following procedure: To calculate the smoothed value for reading number n, perform a least squares quadratic regression on readings n-2, n-1, n, n+1, and n+2, using the reading numbers as abscissas and the readings as ordinates. Substitute n into the resulting equation and solve for the smoothed value. This procedure is justified in the publication "Smoothing and Differentiation of Data by Simplified Least Square Procedures" by Abraham Savitzky and Marcel J. E. Golay.[4,5]

Figure 4 is a flowchart of the peak height determination procedure. As that figure shows, the BASIC program will first access the sixty readings which were taken from the analogue to digital converter by the assembly language routine. The sixty readings will be searched for a place that is level, as defined previously. If no level place can be found, the program will not attempt to determine a peak height and the sample will be logged for rerun. If there are several readings in level places, the program will compare their smoothed values and will locate the highest level reading.

Next, the program will decide which of two peak height determination algorithms to use. The peak heights of "up and over" peaks (such as those shown in Figures 3A and 3B) will be determined by one algorithm, while the heights of all other peaks (such as those shown in Figures 3C and 3D) will be determined by a second algorithm. The program will distinguish between the two classes of peaks by checking whether the top of the peak is concave downward. Mathematically, this will be done by checking whether the smoothed value of the highest level reading is higher than the smoothed values at both of the endpoints of the data acquisition window. The third and fifty-eighth readings will be used as the endpoints of the window because they are the first and last readings for which smoothed values can be determined.

If the peak in question with is an "up and over" peak, it will satisfy the condition of being higher at its highest level reading than it is at either endpoint. Its peak height will be taken to be the smoothed value of the highest level reading.

For peaks which do not go "up and over," the highest level reading is not always the best indication of peak height. For example, in Figure 3C, the highest level reading would be found before the plateau, at the first

reading which satisfies the definition of "level," while the best place to measure the peak height would be on the plateau. Therefore, if the peak in question is not an "up and over" peak, the program will determine the peak height simply by examining the data at the expected position of the most valid reading. The expected position will have been predicted using an algorithm described in the next section. If the expected position of the most valid reading is not level, the program will not attempt to determine the peak height. If the expected position is level, the peak height will be taken to be the smoothed value of the reading at the expected position.

4.3 SYNCHRONIZATION WITH THE AUTOMATIC SAMPLER

Timing synchronization involves keeping the data acquisition windows centered on the peaks and predicting the expected positions of the most valid readings. The timing synchronization algorithms to be used in the Continuous Flow Analyzer Automation System will all be based on a philosophy of "when in doubt, do not change the timing parameters." Connectors A and B show how the peak height determination algorithms in Figure 4 will be linked to the timing synchronization algorithms in Figure 5.

If the height of a peak is not determined by the highest level reading algorithm, that peak will have no effect on the timing parameters. That is, the dead time between data acquisition window will not be adjusted, and the prediction for the expected position of the most valid reading in the next peak will remain the same.

If the highest level reading algorithm has been applied, the peak may influence the data acquisition window position, but only if it satisfies all four of the following conditions:

1) The height of the peak will have to be within the range of the heights of the non-zero calibration standards. It would be pointless to make a window adjustment based on a peak of atypical height.

2) The top of the peak will have to be concave downward enough that the highest level reading is at least two times the allowable standard deviation for levelness above the average height of the endpoints. This restriction will prevent a peak such as that shown in Figure 3E from causing a window adjustment.

3) The highest level reading will have to be either before the twentieth reading or after the fortieth reading. As long as the highest level reading is in the central third of the data acquisition window, it will be considered safely centered.

4) The peak must not be the one immediately following a peak which mandated a window adjustment. If peak number n mandates a window adjustment, the BASIC program will instruct the assembly language routine to shorten or lengthen the dead time between two data acquisition windows when it next begins a dead time period. However, the dead time between peak n and peak n+1 will already be in progress. Therefore, the assembly language routine will not implement the window

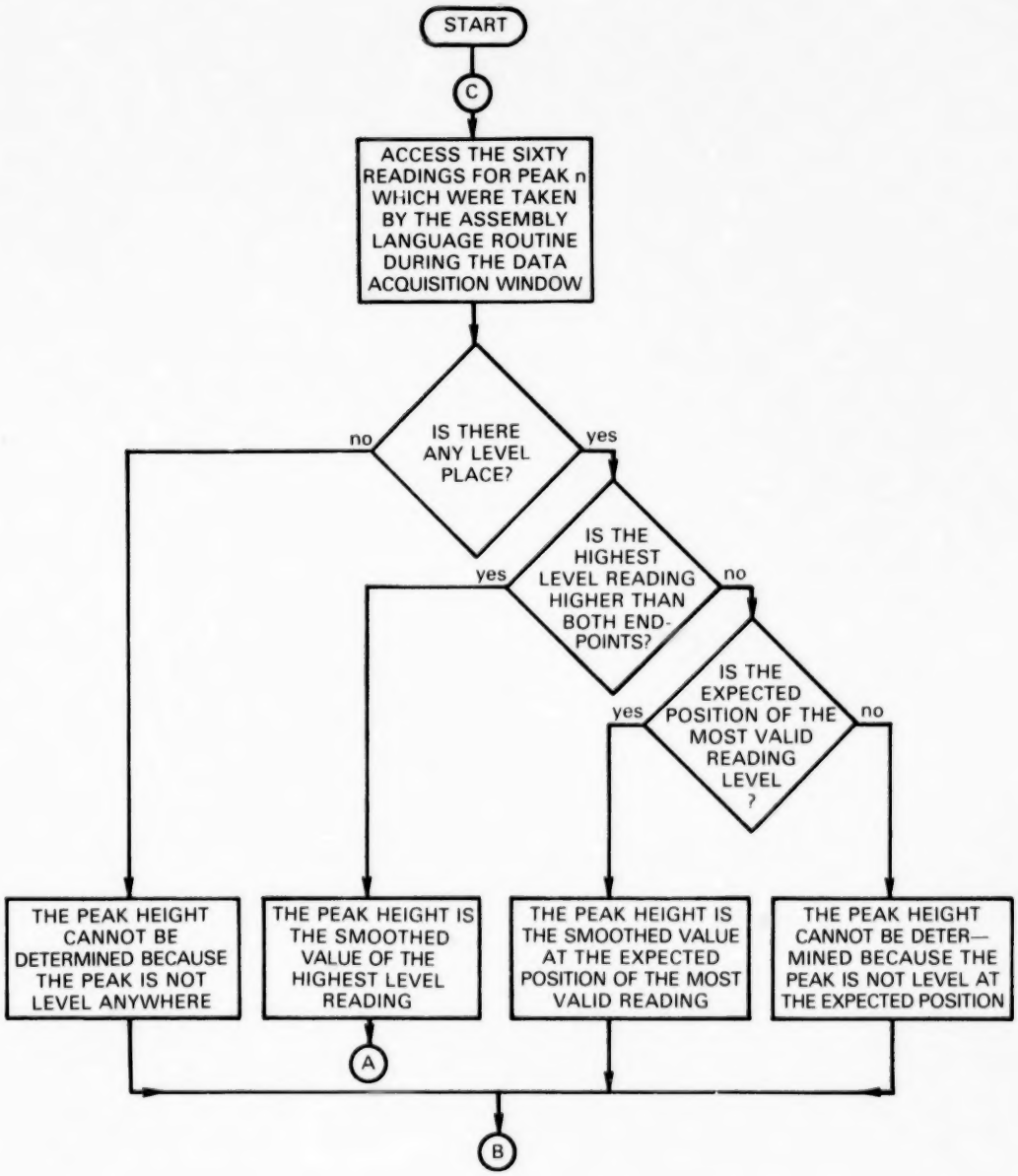
START
C
ACCESS THE SIXTY READINGS FOR PEAK n WHICH WERE TAKEN BY THE ASSEMBLY LANGUAGE ROUTINE DURING THE DATA ACQUISITION WINDOW
IS THERE ANY LEVEL PLACE?
no
yes
IS THE HIGHEST LEVEL READING HIGHER THAN BOTH END-POINTS?
yes
no
IS THE EXPECTED POSITION OF THE MOST VALID READING LEVEL ?
yes
no
THE PEAK HEIGHT CANNOT BE DETERMINED BECAUSE THE PEAK IS NOT LEVEL ANYWHERE
THE PEAK HEIGHT IS THE SMOOTHED VALUE OF THE HIGHEST LEVEL READING
THE PEAK HEIGHT IS THE SMOOTHED VALUE AT THE EXPECTED POSITION OF THE MOST VALID READING
THE PEAK HEIGHT CANNOT BE DETER-MINED BECAUSE THE PEAK IS NOT LEVEL AT THE EXPECTED POSITION
A
B

Figure 4. Peak height determination algorithms.

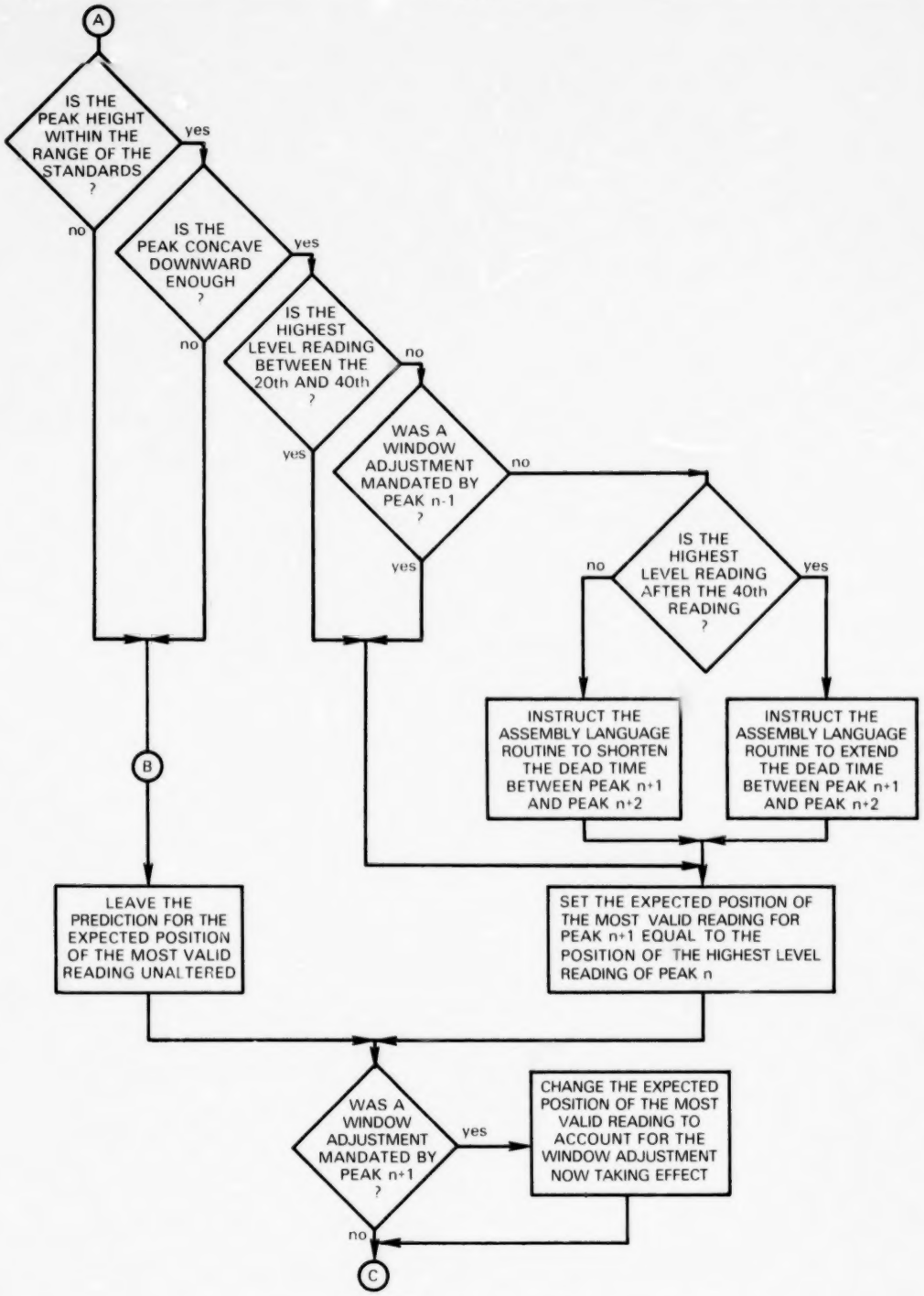
A
IS THE PEAK HEIGHT WITHIN THE RANGE OF THE STANDARDS ?
yes
no
IS THE PEAK CONCAVE DOWNWARD ENOUGH ?
yes
no
IS THE HIGHEST LEVEL READING BETWEEN THE 20th AND 40th ?
no
yes
WAS A WINDOW ADJUSTMENT MANDATED BY PEAK n-1 ?
no
yes
IS THE HIGHEST LEVEL READING AFTER THE 40th READING ?
no
yes
INSTRUCT THE ASSEMBLY LANGUAGE ROUTINE TO SHORTEN THE DEAD TIME BETWEEN PEAK n+1 AND PEAK n+2
INSTRUCT THE ASSEMBLY LANGUAGE ROUTINE TO EXTEND THE DEAD TIME BETWEEN PEAK n+1 AND PEAK n+2
B
LEAVE THE PREDICTION FOR THE EXPECTED POSITION OF THE MOST VALID READING UNALTERED
SET THE EXPECTED POSITION OF THE MOST VALID READING FOR PEAK n+1 EQUAL TO THE POSITION OF THE HIGHEST LEVEL READING OF PEAK n
WAS A WINDOW ADJUSTMENT MANDATED BY PEAK n+1 ?
yes
CHANGE THE EXPECTED POSITION OF THE MOST VALID READING TO ACCOUNT FOR THE WINDOW ADJUSTMENT NOW TAKING EFFECT
no
C

Figure 5. Timing synchronization algorithms.

adjustment until the dead time between peak n+1 and peak n+2. Consequently, a window adjustment mandated by peak n will not take effect until peak n+2. Thus, peak n+1 must not be permitted to mandate another window adjustment.

If a peak satisfies all of these conditions, a window adjustment will be made. If the highest level reading is earlier in the window than the twentieth reading, the dead time between peaks n+1 and n+2 will be shortened. The amount of time by which it is shortened will be enough so that if peak n+2 is identical to peak n, its highest level reading will occur either ten readings later or at the twentieth reading, whichever involves the least change. Similarly, if the highest level reading is later than the fortieth reading, the dead time between peaks n+1 and n+2 will be lengthened enough to cause the highest level reading of peak n+2 to be either ten readings earlier or at the fortieth reading, whichever involves the least change. The dead time will revert to its normal length between peaks n+2 and n+3 and thereafter.

Next, the program will predict the expected position of the most valid reading. To influence this prediction, an "up and over" peak will have to satisfy only the first two of the four conditions above. The prediction will be based on the assumption that, for an "up and over" peak, the highest level reading corresponds to the time when the sample passing through the colorimeter is least diluted by wash solution and least contaminated by other samples. Assuming that the automatic sampler will aspirate samples at regular intervals, the expected position of the most valid reading for the next peak will be set equal to the position of the highest level reading of the current peak.

The program will make one final check regarding the expected position of the most valid reading. If a window adjustment was mandated by the previous peak, the program will alter the expected position to account for the fact that the window adjustment will go into effect on the next peak. This final check will be done regardless of the shape of the current peak.

The program will then instruct the assembly language routine to provide the sixty readings for the next peak.

Naturally, experimentation with these peak height determination and timing synchronization algorithms has proven that they function best with well defined peaks, although they can accommodate occasional merged peaks. However, the alogrithms cannot be expected to perform dependably when confronted with a long series of merged peaks. For this system, the sample-to-wash ratio of the automatic sampler will have to be set low enough to produce peaks which are separate and distinct, although it will not be required that the detector return all the way to the baseline between samples.

4.4 BASELINE DRIFT AND CORRECTION

On a stripchart recording for a batch, the baseline is drawn as a series of straight line segments joining the "peaks" for blanks, and the

peak heights of all other peaks are measured from that baseline. Similarly, in the continuous flow analyzer automation system, the height of the baseline will be interpolated between the peak heights of blanks, and this value will be subtracted from the heights of the peaks for other samples.

Each batch of samples will begin with a blank (after the timing set standards) end with a blank, and have blanks at regular intervals. The automation system will determine the peak heights for the blanks using the normal peak height determination algorithms. If the analyst chooses to place two blanks consecutively, the automation system will determine the peak heights of both blanks, and will use the lower of the two to establish the baseline.

For the final report, peak heights will be calculated relative to the baseline interpolated between blanks. The height of the baseline for a specific sample in an interval between blanks will be calculated by the formula:

$$\text{Baseline height} = \left(\frac{B_2 - B_1}{n + 1}\right)(x) + (B_1)$$

where B_1 is the peak height of the blank before the interval, B_2 is the peak height of the blank after the interval, n is the number of samples in the interval (not counting the blanks), and x is the position of the sample in the interval.

4.5 CALIBRATION

Calibration is the process of developing an equation relating peak height to concentration. The continuous flow analyzer automation system will allow for calibration using from two to ten standards. The analyst will enter the prepared concentrations of the standards for each channel, and the automation system will measure the peak heights. A separate calibration equation will be developed for each channel, although all channels will have to have the same number of standards in the sample wheel. The calibration standards will always come at the beginning of a batch of samples.

4.5.1 Curve Fitting Algorithms

The calibration equations will be derived from the standards information by one of the following techniques: 1) linear regression, 2) quadratic regression, 3) cubic regression, or 4) linear interpolation. Linear regression and linear interpolation will require at least two non-equal standards. Quadratic regression will require at least three standards; cubic regression will require at least four. In each case, at least one standard more than the minimum will be strongly recommended. The analyst will be permitted to specify different curve fitting techniques for different channels. Regression will be performed using conventional least squares techniques.[6]

When a calibration equation is determined, the automation system will also calculate a "fitting error" as an objective measure of the goodness of fit. This fitting error will actually be a percent, relative standard error of estimate, calculated by the following formula:

$$\text{Fitting Error} = \frac{(100)\sqrt{\frac{\sum(\text{Known conc.} - \text{Calc. conc.})^2}{\text{Number of standards} - (\text{Degree} + 1)}}}{\text{Mean conc.}}$$

For any calibration, the automation system will also provide the fitting error for a linear, least squares regression forced through the origin. If the fitting error of the calibration curve being used is reasonable, but the fitting error of the forced origin fit is not, then the analyst can conclude that the calibration curve is abnormally contorted -- a condition which can be explored further by plotting the calibration curve.

4.5.2 Calibration Curve Manipulation

At the beginning of a run, the analyst will specify the type of curve fitting to be used, and immediately after the last calibration standard has been analyzed, the automation system will develop the calibration equations. These equations will be used throughout the run to calculate the concentrations shown on the progress report.

However, after the batch is completely processed, and the system no longer needs to keep up with the automatic sampler, the analyst will have the option of manipulating the calibration curves. It will be possible to delete a standard and/or experiment with other types of curve fitting to try to achieve a better fit. The concentrations printed on the final report and transmitted to the Sample File Control System will be based on the last set of calibration curves generated.

4.5.3 Calibration Curve Plotting

Immediately after calibration is accomplished, the analyst will be able to sign on to a second terminal and obtain plots of the calibration curves while the automation system continues. If the calibration curves are not acceptable, the analyst will be able to abort the run, or allow the run to continue and attempt to improve the calibration equations after the batch has been processed. If the analyst uses the curve manipulation options, it will be possible to plot the new curves also.

4.6 CONCENTRATION CALCULATIONS

The concentration of a sample will be calculated by determining its peak height, subtracting the value for the baseline interpolated between blanks, and substituting this corrected peak height into the calibration equation.

4.7 QUALITY CONTROL CALCULATIONS

The Continous Flow Analyzer Automation System will accommodate five types of quality control samples, namely instrument check standards, laboratory spiked samples, laboratory duplicate samples, laboratory control standards, and laboratory reagent blanks, as defined in the Sample File Control Data Element Dictionary.[7] The system will perform quality control calculations on-line, and it will supply this information on the progress report and the final report. The system will alert the analyst if an out-of-control situation is detected, however, it will be left to the analyst to judge whether the run should be terminated.

4.7.1 Laboratory Spiked Samples

A spiked sample is an actual environmental sample, the concentration of which is measured before and after the addition of a known amount of the analyte being measured. A spiked sample is useful in validating the accuracy of a measurement. It is evaluated on the basis of how nearly the measured increase in concentration corresponds to the known increase.

The following three data elements will be reported for each spiked sample: 1) the laboratory spike original concentration (LSO), which is the measured concentration of the environmental sample, 2) the laboratory spike final concentration (LSF), which is the measured concentration of the spiked sample, and 3) the laboratory spike added value (LSA), which is the amount by which the concentration of the sample will increase due to the addition of the spike.

Most analysts will spike with a minute amount of spiking solution which does not significantly change the volume of the sample. In such a case, the user will be required to enter the volume of the aliquot being spiked and the mass of the analyte in the spiking solution. The system will calculate the LSA value by the formula:

$$LSA = \frac{M}{V_o}$$

where M is the mass of the analyte in the spike and V_o is the volume of the aliquot.

Other users may prefer to spike with significant volumes of spiking solution. Such users will be required to enter the volume of the aliquot being spiked, the volume of the spiking solution, and the concentration of the spiking solution. The system will calculate the LSA value by the formula:

$$LSA = \frac{C_s V_s - C_o V_s}{V_o + V_s}$$

where C_s is the concentration of the added spiking solution, V_s is the volume of the added spiking solution, C_o is the measured concentration of the original environmental sample, and V_o is the volume of the original sample.

The percent recovery for spiked samples will be calculated by the formula:

$$\text{Percent Recovery} = \left(\frac{LSF - LSO}{LSA}\right)(100)$$

The limits for acceptable spiked samples will be expressed in terms of percent recoveries, and will be derived from the measurements for spiked samples in similar previous batches. The upper and lower warning limits will be the mean percent recovery plus and minus two standard deviations. The upper and lower control limits will be the mean percent recovery plus and minus three standard deviations. These limits will not be calculated by the Continuous Flow Analyzer Automation System, but rather will be supplied by either the Sample File Control System or the analyst.

4.7.2 Laboratory Duplicate Samples

Duplicate samples are two test tubes filled with the same environmental sample, and the comparison of their measured concentrations is an indication of the precision of the analytical method. The two reportable values for a duplicate pair are the laboratory duplicate first member (LD1) and the laboratory duplicate second member (LD2).

Duplicate samples will be evaluated in terms of their "range," that is, the difference between their measured concentrations. At low levels of concentration, only a small range will be acceptable, but at higher concentrations, larger ranges will be equally acceptable.

The control limit for duplicates will be the "critical range" value, that is the difference which is considered too great for the concentration level. The critical range values used by this automation system will be derived from the expression:

$$\text{Critical Range} = CX + D$$

where X is the mean measured concentration of the duplicates, and C and D are numbers supplied by the Sample File Control System or the analyst. The values of C and D will be determined from the results of duplicate samples in previous batches such that the above formula will produce critical range values which are three standard deviations greater than the mean range at every concentration level. The values of C and D will not be calculated by the Continuous Flow Analyzer Automation System, but rather will be supplied by either the analyst or the Sample File Control System.

4.7.3. Laboratory Control Standards

Laboratory control standards are solutions of known concentration which are handled like environmental samples in every way. The degree to which the measured concentration matches the known concentration is a reflection of the quality of the results of the run. A control standard will be evaluated by its percent recovery, calculated by the formula:

$$\text{Percent Recovery} = \left(\frac{LCM}{LCT}\right)(100)$$

where LCM is the laboratory control standard's measured concentration, and LCT is the laboratory control standard's theoretical concentration. The warning and control limits for laboratory control standards will have the same form as those for spiked samples, and they will have to be supplied by either the analyst or the Sample File Control System.

4.7.4 Instrument Check Standards

Instrument check standards are replicates of calibration standards. They are used to monitor instrument stability. Each check standard will be evaluated in terms of how nearly its peak height matches that of the calibration standard with the same prepared concentration. This evaluation will be expressed as a percent recovery calculated by the formula:

$$\text{Percent Recovery} = \left(\frac{\text{Peak height of check standard}}{\text{Peak height of calibration standard}}\right)(100)$$

Control limits for instrument check standards will be of the form 100%±n%, where n is an arbitrary value specified by the analyst at the beginning of the run.

4.7.5 Laboratory Reagent Blanks

Laboratory reagent blanks are solutions which contain none of the analyte being measured, but are processed as environmental samples. The measured concentration of a laboratory reagent blank (the LRB value) is an indication of any contamination or interference which may have been introduced by the sample processing. A laboratory reagent blank is evaluated by comparing its measured concentration to a set of control limits which have been derived from the results of previous batches. The upper and lower warning limits will be two standard deviations above and below the mean concentration, the upper and lower control limits will be three standard deviations above and below the mean. All of the limits will be expressed in units of concentration, and they will have to be supplied by the analyst or the Sample File Control System. The possibility of subtracting laboratory reagent blanks is discussed in a later section of this document.

4.8 COMPENSATION FOR DILUTION

Many environmental samples need to be diluted in order to reduce their concentrations to a level within the range of an analytical method. The measured concentration of such a sample must be adjusted so that the reported concentration is that of the original sample rather than the diluted sample. For the automation system to make this adjustment, the analyst will have to enter the volume of the original sample and the volume of the dilution water. The system will calculate the concentration of the original sample by the formula:

$$C_o = C_m \left(\frac{V_o + V_d}{V_o} \right)$$

where C_o is the calculated concentration of the original sample, C_m is the measured concentration of the diluted sample, V_o is the volume of the original sample, and V_d is the volume of the dilution water.

Dilution procedures are complicated somewhat when diluted samples are involved in quality control sets. Obviously, in the case of duplicate samples, both members of the duplicate pair must be diluted identically, and the system will use the formula above to calculate the values reported as LD1 and LD2.

The formula above will also be used to calculate the values for LSO and LSF in cases of diluted samples which are spiked with an insignificant volume of spiking solution. The system will require that dilution be done before spiking. The value for LSA will be calculated by the formula:

$$LSA = \frac{M}{V_o}$$

where M is the mass of the analyte added in the spike and V_o is the volume of the original, undiluted sample.

For analysts who prefer to use spikes with significant volumes, the following formulas must be used to calculate the relevant values for spikes of diluted samples:

$$LSO = C_m \left(\frac{V_o + V_d}{V_o} \right)$$

where C_m is the measured concentration of the diluted but unspiked aliquot, V_o is the volume of that aliquot before dilution, and V_d is the volume of the dilution water.

$$LSF = C_m \left(\frac{V_o + V_d + V_s}{V_o + V_s} \right)$$

where C_m is the measured concentration of the diluted, spiked aliquot, V_o is the volume of that aliquot before dilution or spiking, V_d is the volume of the dilution water, and V_s is the volume of the added spiking solution.

$$LSA = \frac{C_s V_s V_o - C_m V_o V_s - C_m V_s V_d}{V_o V_o + V_o V_s}$$

where C_s is the concentration of the spiking solution, C_m is the measured concentration of the diluted but unspiked aliquot, V_s is the volume of the spiking solution, V_o is the volume of the undiluted,

unspiked aliquot, and V_d is the volume of the dilution water.

A detailed justification for these formulas can be found in the memorandum, "Proposed Sample Dilution Procedures and Sample Spiking Procedures for Sample File Control," by Dennis P. Ryan.[8]

4.9 REMEASUREMENT OF QUESTIONABLE SAMPLES

4.9.1 Peaks to Which the Algorithms Cannot Be Applied

There are two types of peaks to which the peak height determination algorithms cannot be applied: 1) peaks which do not have any level place (e.g. noisy peaks), and 2) peaks which are not "up and over" peaks and which are not level at the expected position of the most valid reading (e.g. shoulder peaks which do not have plateaus). If either of these types of peaks is detected, the system will instruct the analyst to move the questionable sample to the end of the batch, and it will be remeasured.

Both of these types of questionable peaks are identified by their lack of levelness, and the definition of levelness will be specified by the operator. Thus, if the operator specifies a very small standard deviation to define levelness, any peak without a perfectly flat plateau will be rejected. Conversely, if the operator specifies an absurdly high standard deviation to define levelness, no peak will ever be rejected. A few experimental runs should enable the operator to "fine tune" this aspect of the system.

4.9.2 Offscale Samples

If the peak for a sample is significantly higher than that of the highest calibration standard, the Continuous Flow Analyzer Automation System will instruct the operator to dilute the sample and place it at the end of the batch for remeasurement. The actual definition of what constitutes an offscale sample will, however, be an operator-specified parameter. Thus, the operator might specify that the system should force the dilution and remeasurement of any sample whose peak is at all higher than that of the highest standard. Alternatively, it will be possible to set the limiting height absurdly high, and the system will never force the remeasurement of concentrated samples.

4.9.3. Operator-Detected Unacceptable Data

Occasionally, an operator may wish to remeasure a sample even though the automation system has not detected any reason for forcing a remeasurement. However, the system cannot account for samples which are moved to unknown positions in the sample wheel. Therefore, at the beginning of an automated run, this system will allow the operator to designate some sample wheel positions at the end of the batch as "extra repeat positions." These positions in the sample wheel will normally be filled with blanks, but if, in the judgment of the operator, a sample must be remeasured, it will be placed into one of these positions. The peak heights and concentrations of all of the repeat positions will be reported normally on the final report,

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and it will be the responsibility of the operator to remember the identity of each repeat position. A feature which can be used after the batch has been completely processed will provide a manual method for associating the concentrations of repeat positions with the correct SFC identification numbers.

4.10 REAGENT BLANK SUBTRACTION

Laboratory reagent blanks can be used not only as a quality control evaluation, but also as a way of correcting for contamination and interference introduced by the sample processing. This automation system will enable the analyst to subtract the instrument signal value for a laboratory reagent blank from that of the environmental samples after the automated run has been completed.

4.11 IDENTIFICATION OF CONCENTRATIONS BELOW THE DETECTION LIMIT

Some users of this system will want to identify those samples whose concentrations are too low to be legitimately measured by the instrument. Therefore, the system will allow the user to enter a detection limit in terms of concentration, and any measurements below that limit will be shown on the final report and transmitted to the Sample File Control System labeled as "below detection limit." The automation system will not calculate the detection limit; it will be determined by the analyst.

The concentrations of reagent blanks will always be shown as calculated and will not be reported as below the detection limit.

4.12 UNATTENDED OPERATION OF THE SYSTEM

The continuous flow analyzer automation system will be capable of operating without human intervention. The analyst will be required to load the automatic sampler and initiate the automated run. Then the system will be able to synchronize itself with the automatic sampler, calibrate, calculate all concentrations and quality control statistics, print reports, and sign off from the computer without manual input. Obviously, the ability to remeasure samples will be sacrificed during unattended operation of the system.

4.13 PROGRESS REPORTS

During the course of a run, the automation system will provide a progress report by which the analyst can monitor the activity of the system. The results for each sample will be displayed as soon as the sample has been analyzed; however, in a multi-channel situation, no measurements will be reported for a sample until the measurement for the slowest channel has been made.

The progress report will show the peak heights of the calibration standards. When calibration has been completed, the report will show the calibration equation and the fitting error for each channel.

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As additional samples are measured, the peak heights and calculated concentrations will be printed, along with any quality control assessments and instructions for dealing with samples which need to be remeasured. Furthermore, if an out-of-control situation develops, the condition will be documented on the progress report, and an audible signal will call the analyst's attention to the event.

The results printed on the progress report will be accurate enough to allow the analyst to judge whether the run is progressing satisfactorily, but will not be adequate for other purposes. The progress report will not account for calibration curve manipulation, reagent blank subtraction, or baseline correction by interpolation between blanks. The height of the baseline for a sample on the progress report will be taken as the height of the previous blank.

4.14 FINAL REPORTS

When all of the data processing for a batch has been completed, a final report will be printed, showing the peak heights for all samples, corrected for baseline drift by subtraction of the value interpolated between blanks. For each channel, the report will show the calibration equation and the fitting error. All of the calibration data will reflect any post-run manipulation of the calibration curve.

The final report will also show the calculated concentrations of the samples, based on the final version of the calibration curve. The reported concentrations will also account for any reagent blank subtraction. If the analyst has specified a detection limit for the batch, concentrations which are below that limit will be labeled as such.

The results which appear on a final report will exactly match those which will be transmitted to the Sample File Control System. As an option, a list of all of the automation parameters for the run may be included in the final report.

4.14.1 Subtraction for Calculated Results

Some analytical methods require the subtraction of two measurements to produce the desired result. For example, a method for the measurement of nitrate involves measuring the total concentration of nitrate and nitrite on one channel, measuring the concentration of nitrite on a second channel, and subtracting to get a measurement of nitrate alone. To accommodate such methods, the automation system will allow the analyst to designate two channels to be subtracted, and the additional set of results will be printed after the final report. Such calculated results will not be reported to the Sample File Control System, since SFC will perform the necessary subtraction itself.

4.15 SAMPLE FILE CONTROL INTERACTION

The Sample File Control (SFC) System is an elaborate EPA data management system which is extensively defined in other documents.[6] Many

users of the Continuous Flow Analyzer Automation System will receive sample backlog lists from the Sample File Control System, and will transmit their results back to SFC.

The Sample File Control System will enable the user to establish a sample wheel pattern -- that is, a list of standards, unknowns, quality control items, and other samples which will appear in the automatic sampler. SFC will then allow the user to select samples from a backlog list. The ID numbers for the samples selected will be placed into a "run file" as dictated by the sample wheel pattern. The Continuous Flow Analyzer Automation System will access the data in the run file. The user will be given a report showing how to fill the automatic sampler, and the automation system will assume that the samples are in the correct positions.

Some users of the Continuous Flow Analyzer Automation System will be involved in research projects which are not being processed by SFC. Instead of getting the ID numbers of their samples from a backlog list, these users will utilize a feature which will allow them to generate their own ID numbers.

After an automated run has been completed and the final report has been printed, the automation system will place all of the results into a "run results file." The user will revert to the SFC system to edit the run results file and transmit it back to the SFC data base.

4.16 UTILITY FEATURES

4.16.1 Instrument and Interface Checkout

At the beginning of each run, the automation system will instruct the analyst to set each detector to full scale and then back to normal damping. This will allow the system to verify that all of the hardware is connected and functioning properly. This feature will also be available without starting the full automation system so that it can be used for routine checks of connections at installation time.

4.16.2 Restart of an Interrupted Run

If an analyst decides to abort an automated run, it will be possible to stop the automation system gracefully. Once the run has been terminated, there will be three alternatives: 1) purge the automation system of all references to the run, 2) restart the automated run, beginning with the timing set standards but without having to re-enter the details for the run, or 3) get a report on the samples measured up to the time that the run went out of control, as determined by the operator.

For interrupted runs involving Sample File Control, any samples which have not been measured at the time of termination will be reported to SFC without a concentration measurement unless the analyst restarts the run. (The Sample File Control RUNEDIT program will enable the analyst to requeue such samples to the backlog to be measured in a later batch.)

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3/8"
PAGE NUMBER
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4.16.3 Raw Data Plotting

After the completion of an automated run, the analyst will be able to obtain plots of all of the raw data acquired by the system.

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SECTION 5

FUNCTIONAL DESIGN

5.1 DESCRIPTION OF AN AUTOMATED RUN

When a user first logs on to the computer, the Sample File Control Master Command Menu will be presented. This menu will include the options of creating a new run file, entering an automation system, editing a run results file, or sending a run results file to the Sample File Control computer. The user will first select the option of creating a run file, which is a list of the samples to be measured, complete with the associated standards, blanks, and quality control items in the order in which they are to be measured. For the Continuous Flow Analyzer Automation System, every run file will begin with three timing set standards followed by two blanks and the calibration standards. Every run file will end with four blanks. The ID numbers of the unknown samples will be drawn from the sample backlog list provided by Sample File Control.

When the run file has been assembled, the user will return to the Sample File Control Master Command Menu and will choose the option of entering the automation system. This will transfer control of the computer to the Continuous Flow Analyzer Automation System.

For any user who is not involved in Sample File Control, there will be a substitute for the above procedure. There will be programs within the automation system which will create a run file as described above, except that the ID numbers for the unknown samples will not come from Sample File Control, but rather will be typed in by the user or generated by the computer program in numerical order between limits set by the user. Control of the computer will then be passed to the main Continuous Flow Analyzer programs.

Figure 6 shows the proposed programs of the Continuous Flow Analyzer Automation System and their chaining relationships. Immediately upon entering this system, the user will be confronted with the Continuous Flow Analyzer Master Command Menu. This menu will have the following eight options: 1) generate a non-SFC run file, 2) start an automated run, 3) restart an automated run from the point of the interface check, 4) re-enter the system at the beginning of the post-run manipulations, 5) re-enter the system at the point of approving results for transmission to SFC, 6) check the interface, 7) report and plot the raw data, or 8) return to the Sample File Control System.

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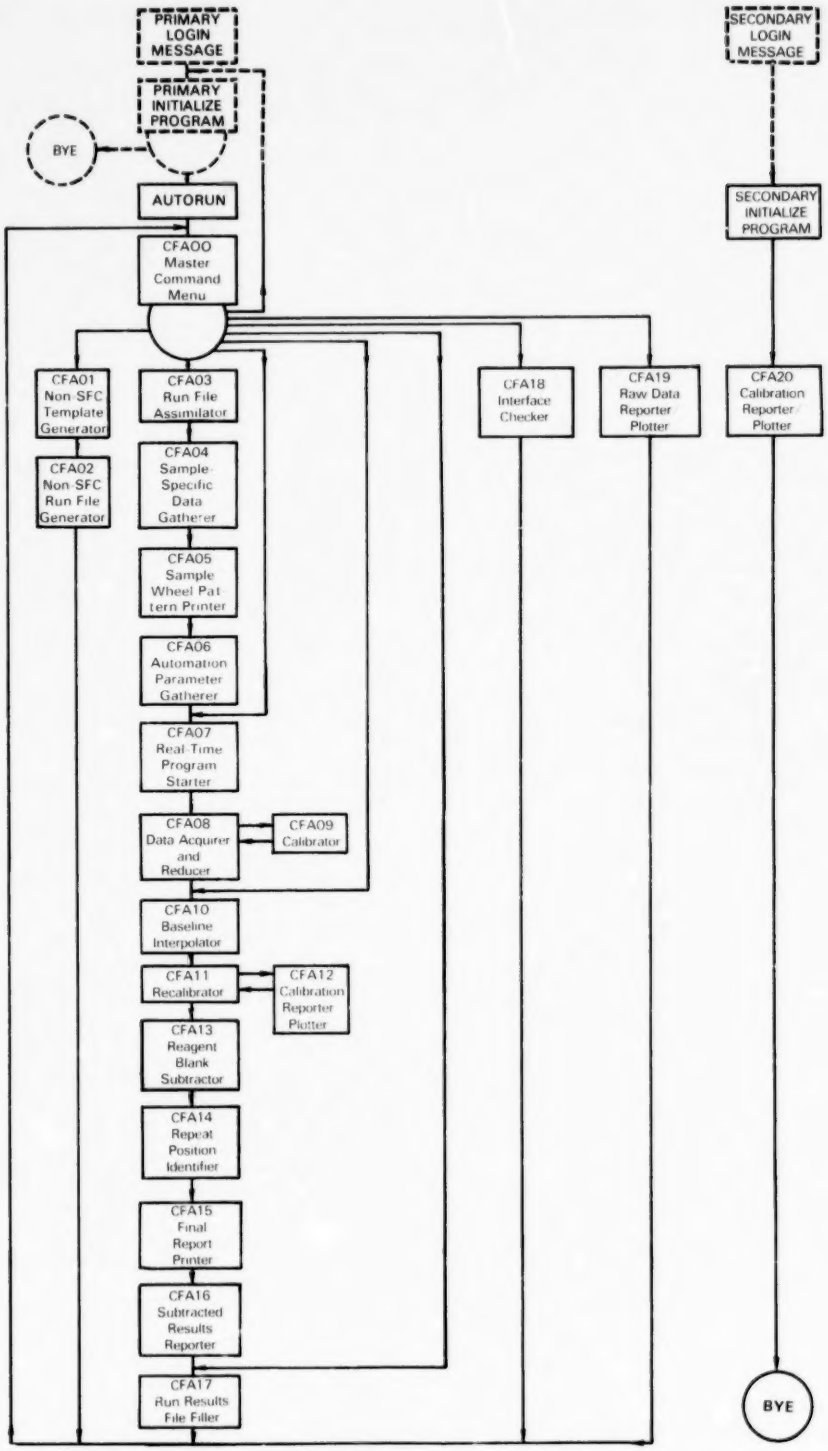

Figure 6. Programs of the Continuous Flow Analyzer Automation System.

If the analyst chooses the second option, the automation system will copy and check the contents of the run file. At this time, the user will enter additional sample-specific data such as the dilution information and the amount of analyte added to spiked samples. Also, the user will be allowed to add to the sample wheel pattern several positions for any samples which he or she may decide to remeasure during the course of the run.

The system will then print the complete sample wheel pattern, unless the user decides to skip this feature. The sample wheel pattern will be a detailed list showing exactly how the sample wheel must be loaded. It will be printed either on the lineprinter or on the user's terminal, at the user's option. Figure 7 shows a typical sample wheel pattern.

The first time the Continuous Flow Analyzer System is used in a directory, the operator will have to enter quite a few parameters including: 1) the sampling rate of the automatic sampler, 2) the number of channels, 3) the data acquisition window width for each channel, 4) the allowable standard deviation for defining levelness for each channel, 5) the upper and lower limits of detection for each channel, 6) the analogue to digital converter channel numbers, 7) the threshold height for the first timing set standard for each channel, 8) the type of calibration curve fitting for each channel, 9) the dilution factors to be used when remeasuring offscale samples, 10) whether the automated run is to be attended or unattended, and 11) what subtraction of results from different channels is to be done, if any. On subsequent runs, the parameters that the operator entered the first time will be used by default unless they are overridden by the operator.

Before the processing of the batch begins, the system will perform a check of the instrument and interface electronics. The user will be instructed to set the detectors to full scale and then back to proper damping, and the computer program will compare the two readings to be sure that they are sufficiently far apart.

The second reason for this check procedure will be to enable the programs to establish the scale for the "percent of full scale." All peak heights will be reported to the user in units where 100 is the instrument response at full scale, and zero is the instrument response for the baseline when the detector is set for proper damping. Thus the user will not need to deal directly with analogue to digital converter counts.

The system will then begin to search for the first timing set standard. The processing of the batch will be completely automatic, and a progress report like that shown in Figure 8 will be printed on the user's terminal. In an attended run, the system will instruct the operator to move any offscale or noisy samples to the end of the batch for remeasurement. In an unattended run, the system will simply not provide measurements for offscale or noisy samples.

As soon as the last calibration standard for every channel has been measured, calibration will be performed using the type of curve fitting specified by the operator earlier. The calibration equations and fitting

S-A-M-P-L-E W-H-E-E-L P-A-T-T-E-R-N
DATE: 05/02/80

WHEEL POSITION	SAMPLE TYPE	DILUTION FACTOR		6210001 RESULTS	6220001 RESULTS	6230001 RESULTS
001 (1/001)	SET STANDARD					
002 (1/002)	SET STANDARD					
003 (1/003)	SET STANDARD					
004 (1/004)	BLANK					
005 (1/005)	BLANK					
006 (1/006)	CAL STANDARD		TCONC:	30.000	15.000	20.000
007 (1/007)	CAL STANDARD		TCONC:	60.000	30.000	40.000
008 (1/008)	CAL STANDARD		TCONC:	90.000	45.000	60.000
009 (1/009)	BLANK					
010 (1/010)	BLANK					
011 (1/011)	ID: 00000001	1.00				
012 (1/012)	ID: 00000002	1.00				
013 (1/013)	SPIKE OF 012	1.00	LSA:	30.000	15.000	20.000
014 (1/014)	DUPE OF 012	1.00				
015 (1/015)	CONTROL STND		LCT:	45.000	22.500	30.000
016 (1/016)	CHECK OF 007					
017 (1/017)	REAGENT BLNK					
018 (1/018)	BLANK					
019 (1/019)	BLANK					
020 (1/020)	ID: 00000003	2.00				
021 (1/021)	ID: 00000004	2.00				
022 (1/022)	SPIKE OF 021	2.00	LSA:	30.000	15.000	20.000
023 (1/023)	DUPE OF 021	2.00				
024 (1/024)	CONTROL STND		LCT:	45.000	22.500	30.000
025 (1/025)	CHECK OF 007					
026 (1/026)	REAGENT BLNK					
027 (1/027)	ID: 00000005	1.00				
028 (1/028)	BLANK					
029 (1/029)	BLANK					
030 (1/030)	REPEAT OF [ ]	1.00				
031 (1/031)	REPEAT OF [ ]	1.00				
032 (1/032)	BLANK					
033 (1/033)	BLANK					

Figure 7. Sample Wheel Pattern.

P-R-O-G-R-E-S-S R-E-P-O-R-T
DATE: 05/02/80

WHEEL POSITION	SAMPLE TYPE	DILUTION FACTOR		6210001 RESULTS	6220001 RESULTS	6230001 RESULTS
001 (1/001)	SET STANDARD		PH:	OK	OK	OK
002 (1/002)	SET STANDARD		PH:	77.448	77.517	77.501
003 (1/003)	SET STANDARD		PH:	76.389	76.437	76.442
004 (1/004)	BLANK		PH:	0.929	0.923	0.942
005 (1/005)	BLANK		PH:	0.912	0.944	0.960
006 (1/006)	CAL STANDARD		BCPH:	32.793	32.728	32.718
			TCONC:	30.000	15.000	20.000
007 (1/007)	CAL STANDARD		BCPH:	62.523	62.568	62.552
			TCONC:	60.000	30.000	40.000
008 (1/008)	CAL STANDARD		BCPH:	91.525	91.564	91.548
			TCONC:	90.000	45.000	60.000
	CALIBRATION FITTING ERROR:			0.57%	0.00%	0.00%
	FORCED ORIGIN FITTING ERROR:			2.60%	2.61%	2.61%
THE SECOND-TERMINAL CALIBRATION REPORT/PLOT IS NOW AVAILABLE						
009 (1/009)	BLANK		PH:	0.130	0.147	0.163
010 (1/010)	BLANK		PH:	0.106	0.135	0.136
011 (1/011)	ID: 00000001	1.00	BCPH:	64.022	64.019	64.014
			CONC:	61.997	30.741	41.008
012 (1/012)	ID: 00000002	1.00	BCPH:	43.726	43.735	43.749
			CONC:	41.106	20.481	27.395
013 (1/013)	SPIKE OF 012	1.00	BCPH:	72.081	72.082	72.119
			LSF:	70.826	34.875	46.599
			LSA:	30.000	15.000	20.000
			RECV:	96.40%	95.96%	96.02%
			QC:	OK	OK	OK
014 (1/014)	DUPE OF 012	1.00	BCPH:	43.532	43.481	43.525
			LD2:	40.980	20.354	27.245
			RNG:	0.198	0.127	0.158
			QC:	OK	OK	OK
015 (1/015)	CONTROL STND		BCPH:	48.126	48.113	48.154
			LCM:	45.593	22.678	30.348
			LCT:	45.000	22.500	30.000
			RECV:	101.32%	100.79%	101.16%
			QC:	OK	OK	OK
016 (1/016)	CHECK OF 007		BCPH:	64.862	63.997	64.825
			RECV:	102.46%	102.28%	102.35%
017 (1/017)	REAGENT BLNK		BCPH:	0.830	0.762	0.880
			CONC:	-2.645	-0.577	0.489
			QC:	OK	OK	OK
018 (1/018)	BLANK		PH:	0.961	0.945	0.992
019 (1/019)	BLANK		PH:	0.975	0.953	1.005
020 (1/020)	ID: 00000003	2.00	BCPH:	101.102	101.165	101.152
			CONC:	OFFSCALE	100.110	133.249
DILUTE BY FACTOR OF 4.00 FOR TOTAL DILUTION OF 8.00; MOVE BLANK FROM WHEEL POSITION 032 (1/032) TO 034 (1/034); PLACE SAMPLE 00000003 IN WHEEL POSITION 032 (1/032)						
021)	ID: 00000004	2.00	BCPH:	58.3[illegible]	58.485	58.389
	[illegible]KE OF 021	[illegible]				43.692
						581

Figure 8. Progress Report.

errors will be included in the progress report. Furthermore, as soon as calibration has been accomplished, it will be possible for the operator to log on to the computer with a plotting terminal and obtain plots of the calibration curves while the automated run progresses, as shown in Figure 9.

At any time during the automated run, the operator may decide to abort the run by pressing the escape key on the terminal. This will return control to the Continuous Flow Analyzer Master Command Menu. That menu will allow three appropriate options as mentioned above: 1) start the run over completely by re-entering all of the parameters and using the same run file to form a new batch, 2) restart the run from the point of the interface checkout without re-entering any parameters, or 3) jump to the baseline interpolation program to ultimately get a final report on those samples which were successfully measured up to the time that the run was aborted. In the third case, any samples which were not measured would have to be included in another batch.

Once the baseline has been interpolated between the heights of the blanks, the operator will have the option of manipulating the calibration curve. Plots of calibration curves will also be available at this point.

If reagent blanks have been included in the batch, the system will then print a report showing the instrument response values for the reagent blanks, the zero standard, the laboratory control standards, and corresponding calibration standards. Using this information, the operator may specify that an instrument response value be subtracted from the unknowns before their concentrations are calculated for the final report.

If during the run the operator moved any operator-detected unacceptable samples to the repeat positions, it will be necessary to tell the computer programs which sample was moved to which position so that the best measurement can be reported to Sample File Control with its correct ID number.

Next, the final report will be printed either on the user's terminal or on the lineprinter, at the option of the user. A typical final report is shown in Figure 10. In an unattended run, the system will print the final report automatically, but it will not perform any of the more elaborate post-run processing. When the analyst returns, it will be possible to repeat the post-run processing, if necessary, before approving the results for transmission to SFC.

Users who are not involved in Sample File Control will be returned to the Continuous Flow Analyzer Master Command Menu after the final report is printed. For Sample File Control purposes, the user will have several other duties. First, the operator will examine the final report and decide whether the results should be reported to Sample File Control. If the operator denotes that the results are completely unacceptable, control will be returned to the Continuous Flow Analyzer Master Command Menu, and the run can be restarted then or later. If there are only a few results which are unacceptable, the operator will be able to delete them individually by using the run editing features of the Sample File Control programs.

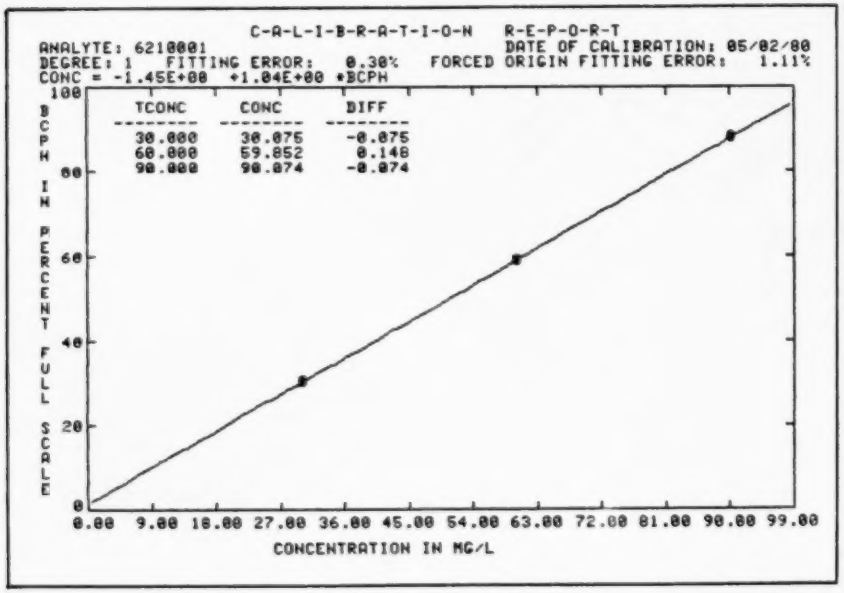
C-A-L-I-B-R-A-T-I-O-N R-E-P-O-R-T
ANALYTE: 6210001
DATE OF CALIBRATION: 05/02/80
DEGREE: 1 FITTING ERROR: 0.38% FORCED ORIGIN FITTING ERROR: 1.11%
CONC = -1.45E+00 +1.04E+00 *BCPH
TCONC CONC DIFF
30.000 30.075 -0.075
60.000 59.852 0.148
90.000 90.074 -0.074
BCPH IN PERCENT FULL SCALE
0 20 40 60 80 100
0.00 9.00 18.00 27.00 36.00 45.00 54.00 63.00 72.00 81.00 90.00 99.00
CONCENTRATION IN MG/L

Figure 9. Calibration curve plot.

F-I-N-A-L R-E-P-O-R-T
DATE: 05/02/80

COMMENTS: SIMULATED THREE-ANALYTE RUN
NAME OF RUN FILE: 6200001.RF
MODE OF OPERATION: ATTENDED
TIME BETWEEN PEAKS: 30 SECONDS
ADDITIONAL DILUTION FACTOR FOR OFFSCALE SAMPLES: 4

	6210001 VALUES	6220001 VALUES	6230001 VALUES
DEFAULT CONCENTRATION UNIT:	MG/L	MG/L	MG/L
A/D CHANNEL NUMBER:	10	11	10
SET STANDARD THRESHOLD HEIGHT:	50	50	50
DATA ACQUISITION WINDOW WIDTH:	20	20	20
STANDARD DEVIATION DEFINING LEVELNESS:	0.60	0.60	0.60
DETECTION LIMIT:	20.000	12.000	10.000
OFFSCALE CONCENTRATION:	90.000	55.000	95.000
ALLOWABLE ERROR FOR CHECK STANDARDS:	10.00%	10.00%	10.00%
TYPE OF FIT:	1	2	0
CALIBRATION FITTING ERROR:	0.38%	0.00%	0.00%
FORCED ORIGIN FITTING ERROR:	1.11%	1.10%	1.12%

CALIBRATION EQUATIONS:
6210001: CONC = -1.45E+00 +1.04E+00 *BCPH
6220001: CONC = -1.13E+00 +5.37E-01 *BCPH -1.41E-04 *BCPH**2
6230001: INTERPOLATION

WHEEL POSITION	SAMPLE TYPE	DILUTION FACTOR		6210001 RESULTS	6220001 RESULTS	6230001 RESULTS
001 (1/001)	SET STANDARD		PH:	100.013	CBD	99.997
002 (1/002)	SET STANDARD		PH:	75.092	75.184	75.136
003 (1/003)	SET STANDARD		PH:	74.788	74.678	74.783
004 (1/004)	BLANK		PH:	1.671	1.689	1.720
005 (1/005)	BLANK		PH:	1.572	1.614	1.644
006 (1/006)	CAL STANDARD		BCPH:	30.302	30.292	30.304
			TCONC:	30.000	15.000	20.000
007 (1/007)	CAL STANDARD		BCPH:	59.929	59.907	59.924
			TCONC:	60.000	30.000	40.000
008 (1/008)	CAL STANDARD		BCPH:	87.983	87.973	87.963
			TCONC:	90.000	45.000	60.000
009 (1/009)	BLANK		PH:	0.275	0.231	0.234
010 (1/010)	BLANK		PH:	0.158	0.171	0.172
011 (1/011)	ID: 00000001	1.00	BCPH:	59.881	SEE 030	SEE 030
			CONC:	60.042		
012 (1/012)	ID: 00000002	1.00	BCPH:	40.722	40.726	40.724
			CONC:	40.913	21.496	27.282
013 (1/013)	SPIKE OF 012	1.00	BCPH:	69.468	69.440	69.444
			LSF:	70.815	35.463	47.245
			LSA:	30.000	15.000	20.000
			RECV:	99.67%	99.78%	99.822
			QC:	OK	OK	OK
014 (1/014)	DUPE OF 012	1.00	BCPH:	42.244	42.213	42.293
			LD2:	42.496	21.272	28.378
			RMS:	1.583	0.776	1.896
			QC:	OK	OK	OK
015 (1/015)	CONTROL STND		BCPH:	44.693	44.694	44.794
			LCN:	45.044	22.578	30.126
			LCT:	45.000	22.500	30.000
			RECV:	100.16%	100.35%	
			QC:	OK		
016 (1/016)	CHECK OF 007		BCPH:	60.815		
			RECV:	101.84%		
017 (1/017)	REAGENT BLNK		BCPH:	0.15		
			CONC:	-7		
			QC:			
018 (1/018)	BLANK		PH:			
019 (1/019)	BLANK					
020 (1/020)	ID: 00000003	2.00				

Figure 10. Final Report.

When the operator approves of the results, the measured concentrations of all of the samples will be placed into the Sample File Control run file, making it into a run results file. It will also be possible to obtain a printout of the run results file at this point, to allow the user to have a hard copy of the quality control limits provided for the batch by SFC. Finally, control will be returned to the Continuous Flow Analyzer Master Command Menu. The only way to leave the automation system will be to return to the Sample File Control Master Commmand Menu. Users not involved with Sample File Control will be able to log off from the computer without going through Sample File Control programs.

Two utility options will also be available from the Continuous Flow Analyzer Master Command Menu. One of these will be to get reports and plots showing the details of the data acquisition process, as shown in Figures 11 and 12. The other utility option will be an interface checkout procedure which will simply allow for checking of hardware during installation.

5.2 PROGRAMS AND THEIR CHAINING RELATIONSHIPS

Figure 6 is a flowchart of the proposed system of programs. The system will operate in a shared directory in which one account ID will allow for access to all of the Sample File Control features and the entire automation system, while the other account ID will allow for access only to the calibration curve plotting program. This arrangement will allow for plotting of calibration curves while the automated run is in progress.

If the escape key is pressed during the execution of any program, control will be transferred back to the Continuous Flow Analyzer Master Command Menu program.

5.3 DATA FILES

5.3.1 Sample File Control Run File

The Sample File Control run file will contain the list of samples to be measured. It will be created by the Sample File Control Foreground NOVA BASIC programs. The data in the run file will be used to initialize the Reduced Data File. The run file will be eliminated when it is transformed into a run results file.

For users not involved with Sample File Control, the Continuous Flow Analyzer Automation System will include an extra program which will create run files. For such a user, a run file will exist until a new run file is created.

5.3.2 Method File

The Method File will contain all of the general user-defined parameters such as the data acquisition window width for each channel, the amount of time between peaks, the threshold height for the first timing set standard, and the degree of the least squares regression for each calibration curve.

R-A-W D-A-T-A R-E-P-O-R-T
DATE: 05/02/80

ANALYTE: 6210001

WHEEL POSITION: 8
PEAK HEIGHT: 88.552
HIGHEST LEVEL POSITION: 33
EXPECTED POSITION: 32

ALGORITHM APPLIED: HIGHEST LEVEL POSITION
TIMING ADJUSTMENT: NONE
HEIGHT AT HIGHEST LEVEL POSITION: 88.552
HEIGHT AT EXPECTED POSITION: 88.545

READING NUMBER	RAW READING	SMOOTHED READING	STANDARD DEVIATION
1	58.794		
2	60.717		
3	63.159	63.881	3.816
4	64.819	64.783	3.227
5	66.236	66.498	3.843
6	69.299	69.357	4.299
7	72.837	72.645	4.116
8	75.804	74.869	3.452
9	76.189	76.553	2.569
10	78.356	77.957	2.432
11	79.214	79.523	2.566
12	81.131	81.085	2.297
13	82.868	82.723	2.188
14	83.727	83.854	1.637
15	84.701	84.653	1.127
16	85.297	85.280	0.835
17	85.579	85.533	0.767
18	85.810	85.941	1.027
19	86.798	86.805	1.081
20	87.798	87.646	0.928
21	87.906	87.952	0.589
22	87.912	87.941	0.149
23	88.040	88.038	0.156
24	88.181	88.169	0.164
25	88.252	88.255	0.137
26	88.316	88.328	0.092
27	88.399	88.377	0.069
28	88.386	88.403	0.048
29	88.418	88.411	0.043
30	88.444	88.450	0.059
31	88.495	88.491	0.063
32	88.534	88.545	0.048
33	88.572	88.552	0.057
34	88.495	88.502	0.082
35	88.418	88.422	0.092
36	88.373	88.373	0.065
37	88.348	88.348	0.038
38	88.335	88.342	0.062
39	88.322	88.343	0.289
40	88.213	88.158	0.543
41	87.668	87.678	0.687
42	87.085	87.141	0.752
43	86.752	86.713	0.709
44	86.383	86.328	0.758
45	85.823	85.815	0.931
46	85.194	85.143	0.969
47	84.387	84.462	0.895
48	83.970	83.972	0.794
49	83.643	83.681	0.752
50	83.806	83.889	0.834
51	82.470	82.494	0.914
52	81.932	81.918	0.910
53	81.330	81.371	0.998
54	80.778	80.716	1.135
55	79.907	79.957	1.314
56	79.073	79.000	1.347
57	78.048	78.135	1.210
58	77.458	77.457	1.138
59	76.920		
60	76.861		

Figure 11. Data Details Report.

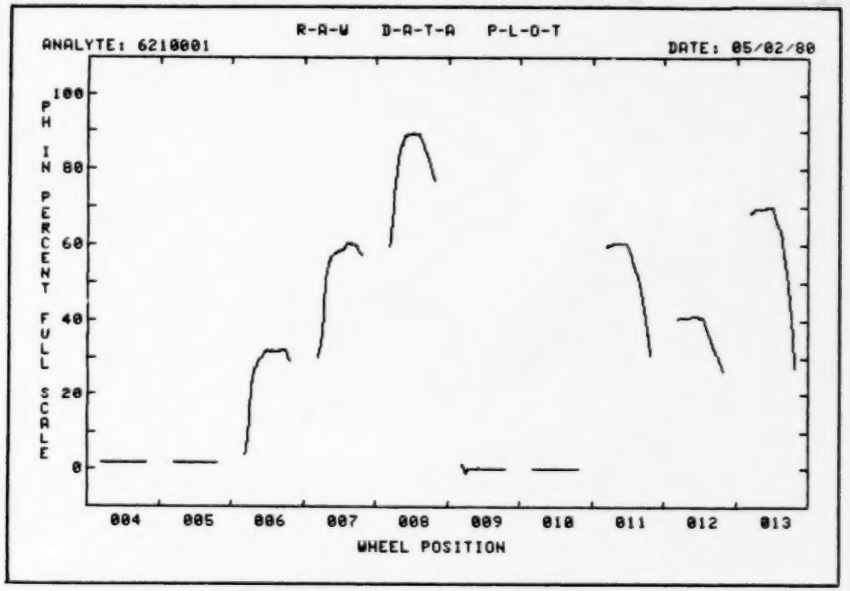

Figure 12. Raw data plot.

The method file will be created the first time the system is used in a directory. It will be filled as the analyst inputs the many parameters for the first time. During the run, the information in this file will be used by all of the programs. In subsequent runs, the parameters in this file will be used by the system as default values, unless the analyst overrides them. When a default is overridden, the new value will be written into the Method File. This file will exist indefinitely.

5.3.3 Reduced Data File

The Reduced Data File will contain the information which is specific to each individual sample such as the dilution factor, the peak height, the quality control limits, and the calculated concentration. One Reduced Data File will suffice for all channels. This file will be created at the beginning of a run, and it will be added to throughout the run. The file will exist until a new Reduced Data File is created.

5.3.4 Raw Data File

The Raw Data File will contain the sixty colorimeter readings and the expected position of the most valid reading for each peak from each channel. It will be created at the beginning of a run, and it will be filled as the run progresses. This file will be used in the raw data details report and the raw data plots. It will exist until a new Raw Data File is created.

5.3.5 Calibration Data File

The Calibration Data File will contain the data necessary for plotting the calibration curves for all channels. The file will be created as soon as calibration is accomplished during the run or when recalibration is done. The file will exist until a new Calibration Data File is created. There may be contention for this file, since it will be used by two programs which may be executed simultaneously.

5.3.6 Sample File Control Run Results File

The Sample File Control run results file will be a run file with the calculated concentrations added to it. All channels will be represented in one run results file. The run results file will be created from the run file after the analyst has approved of the final results. It will exist until it is sent to the Sample File Control System.

For users not involved with Sample File Control, there will be no run results file.

5.3.7 Temporary List File

The Temporary List File will be the file where line printer reports are composed. After the complete report is in this file, it will be copied to the lineprinter, and the file will be deleted. The purpose of this procedure is to minimize the amount of time that the system will monopolize the line printer, which is a shared device.

SIGNOFF SHEET

This document, which describes a proposed continuous flow analyzer automation system but does not constitute an implementation design, is approved by the undersigned interested parties with the understanding that changes in detail are likely and that implementation of all features depends upon the availability of funding and personpower.

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REFERENCES

1. Crawford, R. W. and Barton, G. W. Jr. Technicon AutoAnalyzers; Functional Description, UCRL-52046 (April 1976).

2. Crawford, R. W., Computer Automation of Continuous-Flow Analyzers for Trace Constituents in Water, UCRL-52352 (various dates).

3. Taber, L., Ames., H. S., Yamauchi, R. K., and Barton, G. W. Instrument Calls and Real-Time Code for Laboratory Automation. Lawrence Livermore Laboratory, University of California, Livermore, California, 1978.

4. Savitzky, Abraham, and Golay, Marcel J. E. Smoothing and Differentiation of Data by Simplified Least Squares Procedures. Analytical Chemistry, 36(8): 1627-1639, 1964.

5. Steiner, Jean; Termonia, Yves; and Deltour, Jules. Anal. Chem., 44 No. 11, Sept. 72, p. 1906.

6. Clark, Charles T. and Schkade, Lawrence L. Statistical Methods for Business Decisions. South-Western Publishing Company, Cincinnati, Ohio, 1969.

7. Calculon Corporation. Data Element Dictionary for the Sample File Control System. No. 4370-101. Arlington, Virginia, 1979.

8. Ryan, Dennis P. Proposed Sample Dilution Procedures and Sample Spiking Procedures for Sample File Control. SouthWestern Ohio Regional Computer Center, University of Cincinnati, available from the author through EMSL-Cincinnati, 1979.

9. Almich, Bruce P. EPA Sample File Control Functional Design for the Nova Foreground. United States Environmental Protection Agency, Cincinnati, Ohio, available from the author through EMSL-Cincinnati, 1978.

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